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Effect of nanoprecipitates and grain size on the mechanical properties of advanced structural steels

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ABSTRACT

The microstructure and nanometric precipitates present in advanced structured steel have been studied by high resolution transmission electron microscopy equipped with energy dispersion X-ray microanalysis, in order to relate the nanometric precipitates and grain size with the improvement of the yield strength value of the API steel. The microstructure and nanometric precipitates of the advanced steel were obtained by a combination of thermo-mechanical controlled hot rolling and accelerated cooling procedures. The API steel composition consisted of hot rolled Nb–Ti microalloyed with: 0.07C, 1.40Mn, 0.24Si, 0.020Al, 0.009P, 0.001S, 0.05Mo, 0.5Cr, 0.05Nb, 0.25Ni, 0.10Cu, 0.012Ti, 0.05N in wt%. As a result, this hot rolled steel tested at a strain rate of $5 \times 10^{-3}\ s^{-1}$ showed an improved yield strength from 798 MPa to 878 MPa due to the micrometric grain size of 2.2 μm and to the nanometric precipitates with a size of around 5 nm in the microstructure of the steel studied.

1. Introduction

It has been pointed out that in order to achieve the stringent requirements of advance structural steels, the steel making and plate rolling technologies must be improved for critical applications such as sour service and deep water technologies [1,2]. The new steel making technologies have permitted to produce clean steels with controlled interstitial, macro and micro alloying elements in parts per million.

Plate rolling technologies have adopted thermo mechanical controlled schedules followed by controlled cooling procedures to provide advantages over conventionally rolled steels producing plates with a good combination of strength and toughness [3–5].

Regarding chemistry of pipeline steels, it needs to be designed, such that, it responds to the controlled thermo mechanical processing applied to steel slabs. An appropriate microstructure with their required yield strength and toughness can be obtained through the control of the recrystallization and transformation process during rolling and cooling [6,7].

The increased strength of advance structural steels has been associated with different strengthening mechanism. For instance, grain refinement which is obtained by the addition of small quantities of microalloying elements and by the control of the rolling condition, in this case, both strength and toughness are improved at the same time [8].

Microalloying elements like Nb, Ti, V or Cr introduced into these steels are strong carbide formers and also act as grain refiner while they are in solution in austenite. In amounts near to 0.1% and under properly selected hot working conditions, it can be created dispersive particles of the MX (M–Nb, Ti, V and Cr; X–N and C) phases. Knowledge of the temperatures of beginning and end of precipitation of the MX phases in austenite is very important for designing the hot-working conditions for the microalloyed steels. The presence of nanometric precipitates in steels causes several effects; significantly inhibit the recrystallization of deformed austenite by stopping the sliding of grain boundaries which suppress the transformation of austenite to ferrite and also the nanometric precipitates are fine enough to induce hindrance to mobile dislocations leading to a significant increase in strength [9,10].

The MX phases of various metallic additions have different effect on steel properties. Conventional high strength low alloy steels (HSLA) have been produced at strength levels of up to 560 MPa yield strength by a combination of grain refinement and precipitation strengthening [9,11]. Recently, the hot rolled Nb–Ti microalloyed steels have been developed with yield strength up to 798 MPa, attributed to the high dislocation density, fine-scale precipitation and fine grain size [12]. Therefore, it is important to control the strengthening mechanisms in advanced structural steels to improve the mechanical properties by means of the control of hot rolling conditions and chemical composition.

In this work we present the results on the thermo mechanical controlled rolling and accelerated cooling procedures applied to an advanced structural steel (0.07C, 1.40Mn, 0.24Si, 0.020Al, 0.009P, 0.001S, 0.05Mo, 0.5Cr, 0.05Nb, 0.25Ni, 0.10Cu, 0.012Ti, 0.05N in

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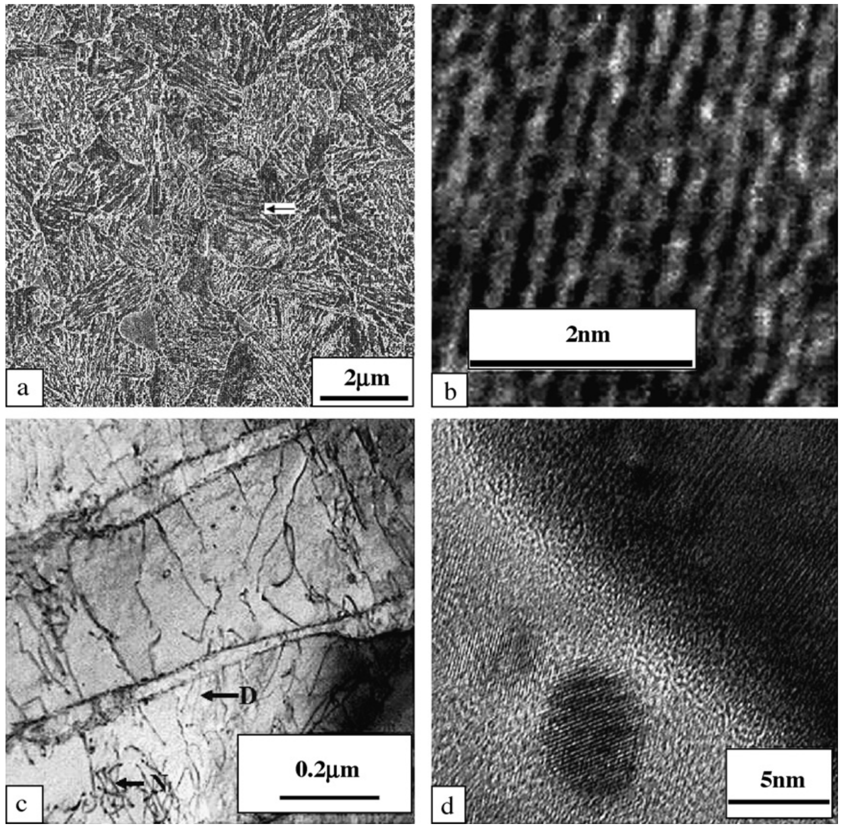

Fig. 1. (a) Packages of martensite sites, (b) HRTEM image of martensite regions, (c) martensite needles (N) with dislocations (D) and (d) HRTEM image of nanometric $Cr_{26}C_6$ precipitates.

wt%) to produce micro grains and nanoprecipitates such that both impact on the strengthening mechanisms which involve grain size, solid solution hardening, dislocation hardening and precipitation strengthening. The use of the steel chemistry responds in this case to the improvement of the yield strength and toughness in advance structural steels.

2. Experimental procedures

The steel under studies was obtained by using electric arc furnace, vacuum degassing, ladle treatment and continuously casting route. The chemical composition of the resulting experimental steel is shown in Table 1. Controlled hot rolling was performed on a Fenn reversible mill. Steel samples were heated at 1250 °C, soaked for 90 min and immediately controlled hot rolled. The rough rolling was performed from 1250 °C to 1098 °C in 5 passes, reaching 42% of deformation. This operation was followed by a cooling period until a temperature of 1050 °C was reached, which corresponds to the temperature of solid solubility of niobium carbonitride in austenite. The finish rolling started at 1051 °C and ended at 867 °C, achieving a deformation of 45% in 5 passes with a strain rate of 8 s^{-1}. After the last pass in finish rolling, the plates were accelerated and cooled, achieving cooling rates of 40 °C/s.

Microstructure of resulting plates was observed in a high resolution transmission electron microscope Jeol 2100 equipped with EDAX microanalysis. Flat tensile (ASTM E-8) tests were conducted on an Instron 1125 (10 ton) test machine at a strain rate of $5 \times 10^{-3}\ s^{-1}$.

3. Results and discussion

Fig. 1 shows the microstructure of hot rolled and accelerated cooled specimens which consist mainly martensite in packet form (Fig. 1a). The lower average of martensitic packets was of 7 μm. Inside, it can be observed the presence of martensite plates with an average length of 2.2 μm and a thickness of 0.33 μm (Fig. 1b). In martensite needles and martensite boundaries, the presence of $Cr_{23}C_6$ carbides (Fig. 1c), with an average size of 5 nm (Fig. 1d) was detected.

Table 2 shows the mechanical properties as function of grain size, where, it can be observed that when the grain size decreases, the yield strength (YS) as well as the ultimate tensile strength (UTS) increases. The maximum experimental yield strength reached in the present work was of 878 MPa, due to small grain size of 2.2 μm and the presence of nanoprecipitates.

Table 1
Chemical composition of steel (wt%).

C	Mn	Si	Al	P	S	Mo
0.07	1.40	0.24	0.020	0.009	0.001	0.05

Cr	Nb	Ni	Cu	Ti	N
0.50	0.05	0.25	0.10	0.012	0.05

Table 2
Mechanical properties of the steel under study.

Grain size (μm)	0.2%YS		UTS		Elongation (%)
	MPa	Ksi	MPa	Ksi	
10	575	83.3	720	104.3	25.2
7	610	88.4	755	109.4	23.4
3	750	108.7	900	130.3	21.5
2.2	877	127.3	983	142.5	16.5

Regarding the strengthening mechanisms for microalloyed steels, the base yield strength (σ_{base}) has been theoretically derived in terms of chemical composition and grain size according to Eq. (1), [13]:

$$\sigma_{base} = \sigma_o + \left[\frac{15.4 - 30C + 6.094}{0.8 + Mn}\right] d^{-1/2} \quad (1)$$

where $\sigma_o = 63 + 23Mn + 53Si + 700P$ gave a base yield stress ($\sigma_{base} =$)$114 + 16d^{-1/2}$.

According to the structure properties analysis, the micrometric grain size of 2.2 μm gave a grain size strengthening of 341 MPa plus the contribution of solid solution hardening of 114 MPa, account for a σ_{base} of 455 MPa.

Regarding dislocation hardening contribution (σ_{disl}), it was estimated using Eq. (2), [12]:

$$\sigma_{disl} = \alpha M G b \rho^{1/2} \quad (2)$$

where α is a constant, M the average Taylor factor, b the magnitude of the Burger's vector, G the shear modulus, and ρ the dislocation density.

Considering typical values for Fe (i.e. $\alpha = 0.3$, $M = 3$, $G = 64$ GPa, and $b = 0.25$ nm) and average $\rho = 9 \times 10^{13}\,m^{-2}$ estimated by determining the number of dislocations in thin regions of the transmission electron microscopic foils of the accelerated cooled sample, dislocation hardening contribution of 130 MPa was obtained.

In addition, the contribution of precipitation strengthening (σ_{pp}) according to De Ardo [14], has been calculated according to the Eq. (3):

$$\sigma_{pp} = \left(\frac{0.538 G b f^{1/2}}{X}\right)\left(\ln \frac{X}{2b}\right) \quad (3)$$

where G is the shear modulus (=81,600 MPa), b the Burgers vector (=0.248 nm), f is the volume fraction of precipitates (=3×10^{-3}) and X is the average diameter of precipitates (=5 nm).

A nanometric carbide precipitation gives a contribution of 275 MPa. However, as has been reported [14], the estimation of contribution to precipitation hardening is not simple.

Taking into account all contributions of the strengthening mechanisms, solid solution strengthening, grain size effect and dislocation hardening, the yield strength value was 860 MPa close to calculated strength of the steel under study and slightly higher than that reported for hot rolled Nb–Ti microalloyed steel [12]. The difference in yield strength values could be due to the contribution of a minor submicron grain size and nanometric precipitates.

4. Conclusion

The chemical composition of the steel under study responded positively to the controlled thermomechanical processing and the applied cooling media allowing reach high values of 878 MPa YS and 983 Mpa UTS. The experimental yield strength obtained in this work was of 878 MPa, mainly due to fine martensite plates with an average length of 2.2 μm and a thickness of 0.33 μm and by the presence of fine precipitates of alloying elements in martensite needles and martensite boundaries with an average size of 5 nm. The experimental yield strength is close to the derived yield strength with a value of 860 MPa and slightly higher to reported in hot rolled Nb–Ti microalloyed steel.

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